National Aeronautics and Space Administration

Computer Automation of Chemical Vapor Deposition (CVD) Chamber for Uniform Growth of Carbon Nanotubes (CNT)

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Introduction

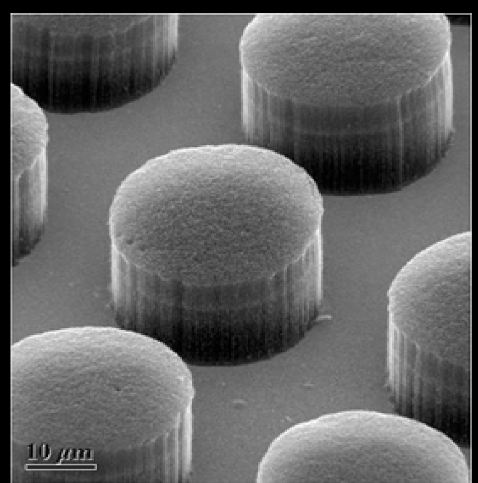

Figure 1 - CNT pillars as viewed from a scanning electron microscope (SEM)

A carbon nanotube (CNT) is a cylindrical nanostructure composed of a one-atom-thick sheet of carbon atoms (graphene). CNTs can be either metallic or semiconducting, with a Young's modulus of at least 1 terapascal and a tensile strength of 200 gigapascal. CNTs are used in a wide range of applications that include atomic force microscopy, clothing, computers, electric circuits, solar cells, and space transportation. While there are several ways to grow CNTs, chemical vapor deposition is the most widespread method, and is used at NASA Ames Research Center.

Chemical vapor deposition (CVD) is a process where CNTs can grow from a substrate coated with a metal catalyst (e.g. iron, nickel, or cobalt), inside a tube furnace reactor. In this process, the metal catalyst is heated up to 750°C, and a process gas and carbon containing gas (e.g. hydrogen and ethylene, respectively) enters the reactor. The carbon containing gas decomposes and the free carbon is reorganized into the CNT structure, which grows from each catalyst particle.

Figure 2 - Quartz tube furnaces

Figure 3 - Control interface for quartz tube furnace

The current CVD furnace (Figure 2) requires the substrate to be placed in the middle of a 1-inch diameter quartz tube. The chamber was controlled by manually turning the knobs of its respective gases and the desired temperature, while a stopwatch was used to keep track of the substrate's CNT growth time. A LabVIEW program interface was created to digitally control the CVD furnace. However, the furnace does not allow a large-scale, uniform growth of CNTs due to its small diameter tube. Therefore, a cold wall hot-plate CVD chamber was built with a similar LabVIEW program interface that digitally controls the gas flow and temperature of the chamber to accomodate CNT growth on larger substrates. While the interface is being used for the furnace and the chamber, the program is being modified to correct any minor bugs and to include additional features users have suggested.

Quartz Tube Furnace

The current CVD method uses a quartz tube furnace, as shown in Figure 2. The quartz tube is either 1 or 1.5-inch in diameter. The process gases (argon, ethylene, and hydrogen) are flowed into the tube from one end, each from their own separate channels. A fourth channel, consisting of argon and a bubbler allows for the incorporation of water vapor during the CNT synthesis.

Before the furnace was connected to the LabVIEW program interface, the entire process was controlled manually (Figure 3). After the substrate is placed in the quartz tube, argon flows through the tube until the temperature reaches 750°C. Argon is used to purge unwanted gases, especially oxygen which will etch or burn the CNTs at such high temperatures. Next, hydrogen is flowed from channel 1 for twenty minutes to reduce the catalyst, and then ethylene is flowed from channel 2 to grow CNTs from the substrate for several minutes. The height is controlled primarily by the length of time that the catalyst is exposed to the precursor, ethylene, and by the temperature of the furnace. Then argon is flowed for five minutes, and the furnace is opened to cool the substrate.

When the furnace was modified to run with the LabVIEW interface, users could grow CNTs in whichever way they wanted instead of following the traditional recipe. However, the size of the substrate is still restricted due to the quartz tube's small diameter. Therefore, the growth of CNTs cannot exceed the quartz tube's diameter. This would make it tedious to grow CNTs on large substrates.

Cold Wall Hot-Plate CVD Chamber

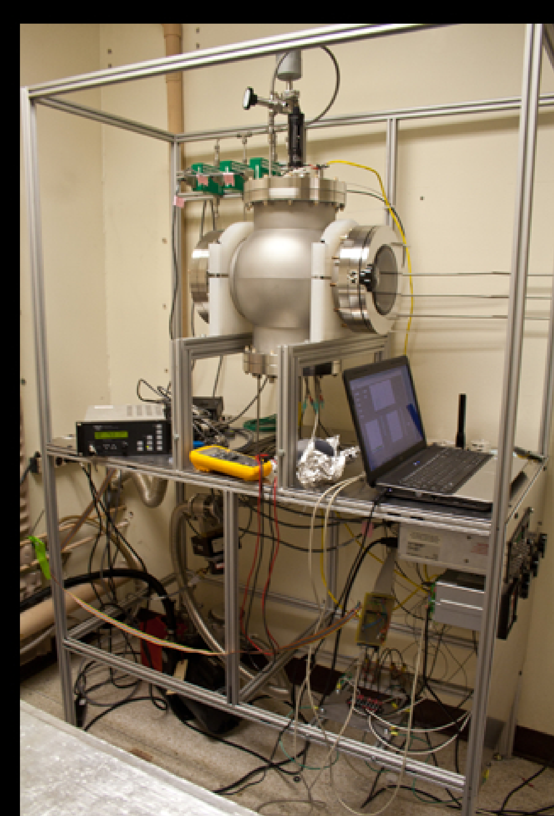

Figure 4 - Cold wall hot-plate CVD chamber

A new CVD reactor was recently built, which uses a cold wall hot-plate CVD chamber, as shown directly to the left (Figure 4). This reactor uses a 6-inch circular hot plate with two heating elements. The substrate is placed on the first heating element, which has a 2-inch radius from the center of the plate. For the substrate to have a more uniform growth of CNTs, a second heating element is used if needed to prevent the loss of heat at the edge of the first heating element. The procedure of growing the CNTs in the chamber is the same as that of the quartz tube furnace.

LabVIEW Program

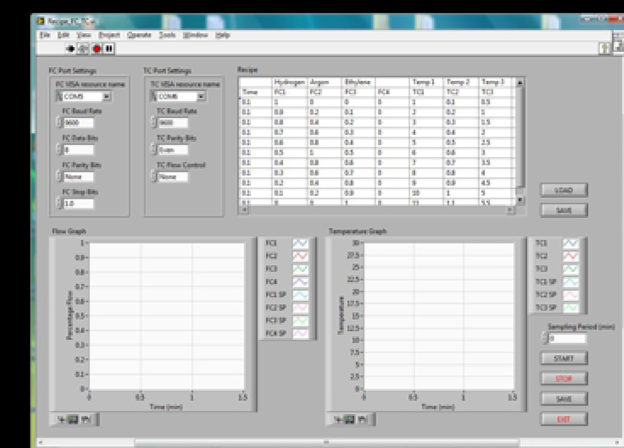

Figure 5 - Front panel of LabVIEW interface

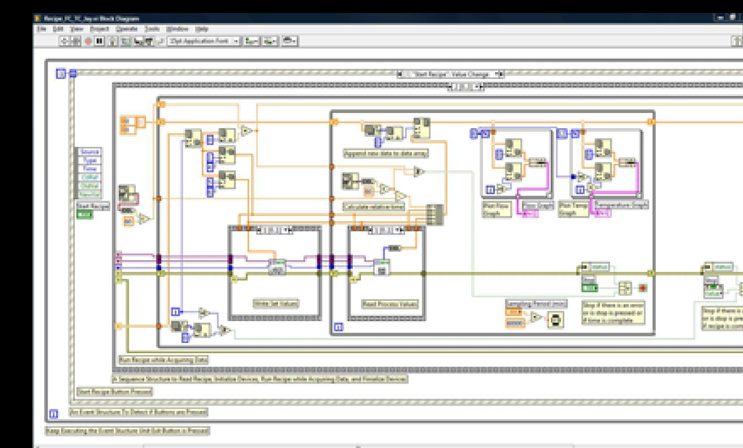

Figure 6 - Block diagram of LabVIEW interface

A LabVIEW program interface was created to digitally control the entire growth process for both the quartz tube furnace and cold wall hot-plate CVD chamber. Figure 5 is a screenshot of the front panel of the interface, where users input or load their recipe for growing the substrate in a sequence of steps. The user configures the flow percentage of each gas, the temperature of the furnace/chamber, and the time limit for each step. Figure 6 shows a screenshot of the block diagram, or the program of the LabVIEW interface.

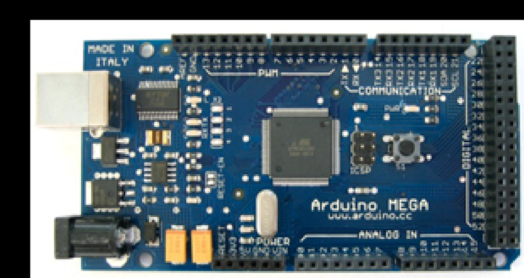

Figure 7 - Arduino Mega microcontroller board

The LabVIEW program interface communicates to the Arduino Mega micro-controller board (Figures 7 and 8). The microcontroller is written in Arduino language, which is loosely based on C/C++. This then relays instructions to a MKS 247D Four Channel Power Supply/Readout, (top right in Figures 3 and 8) which in turn controls the MKS Mass-Flo Controller of each gas or element. Instructions are also simultaneously relayed to an Omega temperature controller (CN7800 for the quartz tube furnace and CN616TC1 for the cold wall hot-plate CVD chamber). This entire process controls and monitors the temperature and gas flow to achieve uniform growth of CNTs.

Figure 8 - (Clockwise from bottom) Arduino board, MKS Mass-Flo Controllers, and MKS Four Channel Power Supply/Readout

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